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TECHNICAL REPORT 3690

**A MODIFIED PICATINNY ARSENAL EXPLOSION
TEMPERATURE TEST FOR DETERMINING
THERMAL SENSITIVITY OF EXPLOSIVES
UNDER CONTROLLED VAPOR EXPOSURES**

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APRIL 1968



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**PICATINNY ARSENAL
DOVER, NEW JERSEY**

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Technical Report 3690

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TABLE OF CONTENTS

	Page
Abstract	1
Introduction	2
Experimental	3
PbN ₆ and RDX	3
Freon-(TF)	3
Water	3
Explosion Temperature Test	4
Calculated Uncertainty of Displacement (t) and Slope (E _a) of Rate Curves	5
Results and Discussion	5
Acknowledgment	8
References	8
Distribution List	22
 Tables	
1 Typical data on times to explosion as a function of T _E (PbN ₆ /vacuum system)	9
2 Percent changes in explosion temperature and activation energy of RDX exposed to vapors and pretreated at 25° and 74° for 72 hours	10
3 Percent changes in explosion temperature and activation energy of PbN ₆ exposed to vapors and pretreated at 25° and 74° for 72 hours	11
4 Percent changes in explosion temperature and activation energy of PbN ₆ /RDX exposed to vapors pretreated at 25° and 74° for 72 hours	12
 Figures	
1 Sensitivity of RDX to thermal explosive decomposition as a function of various exposures at 25°C for 72 hours	13
1A Sensitivity of RDX to thermal explosive decomposition as a function of various exposures at 74°C for 72 hours	14

		Page
Figures		
2	Sensitivity of PbN_6 to thermal explosive decomposition as a function of various exposures at 25°C for 72 hours	15
2A	Sensitivity of PbN_6 to thermal explosive decomposition as a function of various exposures at 74°C for 72 hours	16
3	Sensitivity of PbN_6/RDX to thermal explosive decomposition as a function of various exposures at 25°C for 72 hours	17
3A	Sensitivity of PbN_6/RDX to thermal explosive decomposition as a function of various exposures at 74°C for 72 hours	18
4	Adsorption isotherm, H_2O on RDX at 25°C	19
5	Adsorption isotherm, H_2O on PbN_6 at 25°C	20
6	Assembly unit for the containment of vapors in contact with the explosive during the induction period of thermal explosive decomposition	21

ABSTRACT

The Picatinny Arsenal explosion temperature test has been modified to study the effect produced on the induction period by vapors in contact with the surface of explosives. The test has been found to be reproducible and significantly responsive to changes in times to explosion and activation energy of RDX, PbN_6 , and RDX/ PbN_6 mixture exposed to H_2O and Freon vapors. These changes are postulated to be a function of vapor-surface interactions which correspondingly alter the rate of surface heat dissipation and mechanism of chemical reactions which initiate explosive decomposition.

INTRODUCTION

According to Bowden (Ref 1) and Cook (Ref 2), initiation of pure C-H-N-O explosives is induced by thermal pulses which promote surface hot spots on the order of 400-500°C. Chaiken (Ref 3) further assumed that, on the basis of a mathematical analysis of linear pyrolysis work on the explosives, TNT, Tetryl, RDX and PETN, surface melting by external stimuli such as impact and friction should affect the initial rate of heating of explosives in the condensed phase. Similarly, Cantrell (Ref 4) and Nachbar (Ref 5) have demonstrated the need for considering gas-film effects at the surface of substances undergoing linear pyrolysis. In these studies the surface effects are reported to be endothermic in nature. Thus, induced changes in the rate of surface heat dissipation should produce corresponding changes in thermal sensitivity.

Chaiken (Ref 3) attributed the rate of endothermic surface processes to a surface property and not to the melting point of the explosive in the condensed phase. The question arises as to what particular surface property determines the rate of heat dissipation and hence sensitivity to thermal initiation. From the thermodynamics of water adsorption, the degree of heterogeneity, polar nature and free energy of the surface of explosives can be determined. The capability of determining these surface properties was developed in this laboratory, using beta-HMX as the explosive for study (Ref 6). Accordingly, induced changes in surface properties can be measured and related to observed changes, if any, in sensitivity to initiation.

Before any correlation can be made of surface properties with initiation, a test is required that could demonstrate a measurable response to stimuli affecting the rate of surface heat dissipation, and hence sensitivity to initiation.

Isothermal decomposition has been used to determine bond strengths and activation energies, but does not represent realistic temperatures and rates for explosive decomposition processes. Isothermal decomposition is observed over periods of hours and no explosion takes place. In the friction and impact sensitivity tests explosives are initiated by the conversion of mechanical work to a thermal hot spot which finally grows through a thermochemical mechanism to a runaway reaction or explosion. The adiabatic initiation process associated with this test is subject to many physical variables.

In the explosion temperature test, thermal pulses directly trigger the runaway reactions. Such thermal initiations are on the order of one to ten seconds. Reaction times can be measured conveniently in this time span and kinetic parameters are obtained from realistic condensed phase explosive decomposition. This test is even more useful for measuring induction times to thermal initiation because of its insensitivity to variations in particle size distribution above the minimum critical size of approximately one micron (Ref 7), in which range the particles of most

C-H-N-O explosives are normally found. The explosion temperature test was, therefore, deemed most suitable for this study.

Since sensitivity to initiation can be considered a function of endothermic surface processes, changes in the rate of heat dissipation could produce corresponding changes in times to explosion. One possible manner in which times to explosion could be altered is by gas-surface interactions that would either promote (sensitization) or retard (desensitization) endothermic surface melting during the induction period. Accordingly, when the explosive is sensitized the rate of surface heat dissipation is decreased and when it is desensitized the rate is increased.

The existing PA explosion temperature test required modifications to control various exposure conditions to which explosives could be subjected at the time of thermal initiation to explosive decomposition. This report describes the modifications made and the application of the test in the study of the thermal sensitivity of the Special Purpose grade lead azide and Class A RDX mixture of the "Gravel" type mine exposed to aqueous and nonaqueous vapors of Freon-TF.

The significance of the data obtained in that study has been reported in the Gravel Research Program status report dated 5 October 1967 and is not the subject of this report. Instead, the data generated in that study is used to demonstrate the value of the modified PA explosion temperature to measure changes in the thermal sensitivity of explosives as a function of surface effects.

EXPERIMENTAL

PbN₆ and RDX

Special-Purpose PbN₆ and RDX were dried under vacuum (10^{-6} mm Hg) at 60° for 48 hours. All samples for the explosion temperature test were drawn from the same master batches of PbN₆ and RDX. When the explosives were tested separately, the sample weights were 30 mg, and as a mixture consisted of 17 mg RDX and 11 mg PbN₆.

Freon-TF

The Freon-TF (high purity trichlorotrifluoroethane marketed by E. I. DuPont Co.) was degassed by freeze-thawing under vacuum at -78° prior to use. Pressures of Freon vapors were read with a Wallace & Tiernan gauge.

Water

Distilled water was redistilled from alkaline permanganate and sulfuric acid, followed by a second redistillation. The water was finally degassed by freeze-thawing

under vacuum at -78° . Pressures of water vapor were read with an Apiezon oil manometer having 15 times the sensitivity of a Hg manometer.

Explosion Temperature Test

The PA explosion temperature test apparatus used in this study is essentially a reproduction of the one described by Henkin and McGill (Ref 8). Instead of using cork to consolidate and confine the explosive sample in a No. 8 aluminum blasting cap, three 22-caliber aluminum gas checks were substituted. The commercial analysis of the copper content in the aluminum was stipulated to be less than 0.005%. Aluminum was substituted for the gilding metal (95/5 copper/zinc) normally used, to preclude the formation of copper azides as an intolerable contaminant.

Ordinarily after loading the blasting cap with the sample and gas checks, the contents are arbor-pressed and crimped. However, for those samples to be tested under controlled exposures, the contents were pressed but not crimped at this stage. The uncrimped blasting cap was secured to a pyrex glass flame-seal unit by means of a metalized epoxy cement. The resultant blasting cap-glass assembly was attached to a glass vacuum line manifold by a Swagelok, toggle-valve arrangement. Prior to dosing with vapors, the manifold-sample system was evacuated to 10^{-5} torr over an hour period. In the case of Freon vapors alone, 320 mm pressure was introduced in each of the blasting cap-glass assemblies. For the H_2O -Freon mixture, 6.5 mm H_2O vapor was added to 320 mm Freon. This Freon containing 2% water is referred to as aq-Freon. In one set of the PbN_6 series water was added first, followed by the Freon addition; and in another set the order of addition was reversed as indicated in the respective Tables and Figures. Water alone was dosed at a pressure of 8 torr. To prevent pyrolysis of the vapors when flame-sealing the assemblies, the vapors were condensed previously into the lower portion of the blasting cap with a liquid N_2 bath. For those series requiring heat treatment the sealed assemblies were placed in a constant temperature bath set at $74^{\circ} \pm 1^{\circ}$ for 72 hours.

Electrical continuity across the glass section of the assembly, bridging the blasting cap to a wire secured to the tip of the glass tube for the later hookup, was achieved by an application of conductive silver paste.¹ Before test firing, the blasting cap was finally crimped in the usual manner, and the assembly was then connected to the electric timer via the terminal hook-up wire on the glass tube. The timer started upon immersion into the Wood's metal bath, and was stopped by the ensuing explosion, which shattered the conductive application along with the glass tubing.

¹ The details of the assembly unit are graphically illustrated in Figure 6.

A check was made on the nature of the seal produced by the arbor-pressing of the three gas checks. Six assemblies (previously loaded with an inert material in lieu of explosive and three gas checks and then arbor-pressed) were opened at the base to the atmosphere and attached to the vacuum line. A temporary sealant was placed over the openings and the assemblies evacuated to 10^{-5} torr, holding. The valves to each assembly were closed and the sealant removed. After a lapse of 10 minutes the valves were opened; each of the assemblies registered atmospheric pressure. Apparently, the action of the arbor-pressing of the three gas checks does not hermetically seal the consolidated explosive. In this manner any vapor introduced into the assembly could diffuse to and equilibrate with the explosive.

Calculated Uncertainty of Displacement (t) and Slope (E_a) of Rate Curves

Approximately twelve determinations of times to explosion (t) were made at each of the five temperatures selected for the respective samples. A typical set of data on t 's as a function of explosion temperature is shown in Table 1 (p 9). The data generated in this manner was plotted as $\log t$ vs $T(^{\circ}\text{K})^{-1}$. The respective equations for a straight line (derived by the method of least squares) were used to draw the curves and to calculate the uncertainty of displacement of the curve, which was found, on the average, to be $2 \text{ sigma} = 0.30$ second. The corresponding equation for the standard deviation of the slope yielded an uncertainty of $2 \text{ sigma} = 1.8$ kcal/mole for the activation energy (E_a).

RESULTS AND DISCUSSION

A measure of the sensitivity of an explosive to thermal pulses is the delay time to explosion for a given explosion temperature (T_E). The interval of time between the start of heating and the moment of initiation of explosive decomposition is called the induction time or time to explosion (t). For explosive materials the relationship between t and T_E is expressed by:

$$t = ce^{E_a/RT} \quad (1)$$

where E_a is the activation energy in kcal/mole; c is the constant dependent upon the experimental geometry and the composition of the explosive; T is the explosion temperature in $^{\circ}\text{K}$; and R the usual gas constant.

The relationship given by equation 1 can be conveniently represented in the logarithmic form:

$$\ln t = \ln c + E_a/RT \quad (2)$$

From equation 2 it follows that between $\ln t$ and T^{-1} there exists a linear dependency which has been confirmed by the results obtained in this study and by those reported in the open literature for the majority of explosives investigated. The slope of the straight line is equal to E_a/R . Thus, on the basis of the experimental data concerning T_E , it is possible to determine E_a . Because the entire explosive is not simultaneously subjected to isothermal heating the E_a is an apparent one and this fact is implied whenever E_a is mentioned in this report. However, any dramatic change in E_a induced by various exposures during the initiation period could serve to indicate that the overall mechanism of surface endothermic rate processes is being altered.

From the data plotted in the Figures 1 to 3-A, the percent changes in T_E and E_a were calculated and listed in the Tables 1-3 (pp 9 - 11). Significant changes in T_E are based on the respective displacements in terms of t ($2\sigma = \pm 0.30$ sec) equivalent to $2^\circ K(T)^{-1}$ and those of E_a are related directly to $2\sigma = \pm 1.8$ kcal/mole for the activation energy. All changes in T_E and E_a of the separate and mixed RDX and PbN_6 , as a function of vapor exposure at $25^\circ C$ and $74^\circ C$, were related to the vacuum-exposed samples pretreated at $25^\circ C$.

The T_E at the one-and five-second intercepts of the respective rate curves were selected for the analysis of the data. From equation 1 it is evident that a decrease in t produces a rapid decrease in T_E ; i.e., at the five-second induction periods the explosive with a lower E_a ignites at lower rates of thermal energy input. When the curve of a pretreated sample shifts completely to the left of the control sample (vacuum exposure at $25^\circ C$) and there is an increase in E_a , the explosive is desensitized. In this case both the one-second T_E and five-second T_E are higher than those of the control. Conversely, when the curve is displaced to the right of the control and the E_a is decreased together with decreases in the one-and five-second T_E 's, the explosive is clearly sensitized. In less pronounced examples, these displacements would be parallel to the control, in which case there is no change in E_a . Obviously, intermediate cases of displacements of the rate curve relative to the control are less definitive.

Displacements described by $\% \Delta E_a$ and $\% \Delta T_E$ with the appropriate sign at the one- and five-second induction periods are reflected in Tables 2 to 4 (pp 10 to 12). Therefore, any observed displacements of rate curves of thermal explosive decomposition induced by pretreatment (e.g., by exposure to vapors at elevated temperatures) can serve to illustrate how the rate of surface heat dissipation is being altered.

That the observed changes in T_E and E_a are a function of surface effects is supported by the water adsorption isotherms shown in Figures 4 and 5 (pp 19 and 20). These isotherms illustrate that water adsorption is restricted to the surface of

the substrate explosives. In the event the crystalline structure of either PbN_6 and RDX was sufficiently open to accommodate the water molecule with a diameter of 4.24 Å, the relationship of water sorption with pressure would be monotonic. Since the Freon molecule ($\text{Cl}_2\text{FC}-\text{CF}_2\text{Cl}$) is much larger and less polar than the H_2O molecule, the adsorption, if any, of Freon vapor is necessarily confined to the respective substrate surfaces. Hence, any interaction between these vapors and the explosives must be restricted to the surface, at least during the induction period of thermal explosive decomposition.

The marked effect the vapors have on T_F and E_a is graphically illustrated in Figures 2-3A (pp 15-18). Apparently, this effect is more pronounced with PbN_6 than with RDX; and to a greater extent at elevated temperatures. The greatest changes in T_F (or the induction times) and E_a promoted by the vapor-surface interactions are produced with the mixture PbN_6/RDX heated at 74°C for 72 hours prior to initiation. Heat alone (vacuum) is observed to have a significant effect on the displacement of the curves when compared to the unheated vacuum-exposed sample. Nevertheless, the changes induced by the heated vapor/sample systems are considered real, relative to the vacuum-heated samples. Obviously, the heat treatment complicates any interpretation of the nature of the vapor-surface interactions. In addition, the choice of the vapors and explosive samples, which were dictated by the Gravel Research Program, is not the simple case one would choose to elucidate the fundamental parameters involved in the observed alteration of the surface heat dissipation process. With a more systematic and quantitative approach, however, the nature of the vapor-surface interaction can be determined. It may be possible, for example, to demonstrate that the delays in t 's are a function of desorption of vapors from the substrate explosive, which would be an endothermic surface process. In this case the rate of surface heat dissipation would be increased. Changes in E_a could conceivably be attributed to ensuing reactions between the vapors and the substrate, promoted by conditions of excitation associated with the high temperatures and pressures of the explosion temperature test.

What has been demonstrated by this data is that the modified PA explosion temperature test is effective in measuring changes in t 's and E_a which are produced by the presence of the vapors in contact with the explosives, undoubtedly due to some type of vapor-surface interaction. With such a test the determination and measurement of these parameters can now be studied as a function of induced changes in surface properties.

ACKNOWLEDGMENT

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TABLE 1

Typical data on times^a to explosion as a function of T_E ^b
(PbN_6 /vacuum system)

	340°	345°	355°	360°	370°
	3.0	2.8	2.0	1.8	1.4
	3.3	2.8	2.4	1.6	1.3
	3.3	2.9	2.2	1.5	1.4
	3.2	2.9	2.0	1.5	1.3
	3.1	2.8	1.8	1.9	1.4
	2.8	2.6	2.5	1.8	1.2
	3.1	3.0	1.9	1.4	1.1
	3.3	2.6	2.1	1.5	1.3
	3.8	3.0	2.1	1.7	1.2
	3.2	2.8	1.7	1.8	1.3
	3.3	2.9	1.8	1.6	1.4
	3.6	3.1	2.0	1.5	1.2
\bar{x}	3.2	2.9	2.0	1.6	1.3
s	0.2	0.1	0.2	0.1	0.1

$\log y = -9.575 + 0.062x$, equation derived by least squares for a straight line

^aTime in seconds.

^bIsothermal control, $\pm 1^\circ$.

TABLE 2

Percent changes in explosion temperature and activation energy of RDX
exposed to vapors and pretreated at 25° and 74° for 72 hours

Vapor Exposure	RDX Pretreated at 74° for 72 Hours					RDX Pretreated at 25° for 72 Hours						
	t = 1 sec		t = 5 sec		E _a (kcal/mole)	t = 1 sec		t = 5 sec		E _a (kcal/mole)		
	T _E (°C)	%ΔT _E	T _E (°C)	%ΔT _E		T _E (°C)	%ΔT _E	T _E (°C)	%ΔT _E			
aq-Freon	322	-	267	-	18.3	-	315	-1.3	262	-	18.3	-
H ₂ O	333	4.4	268	-	16.0	-14.9	317	-	264	-	20.2	-
Freon	333	4.4	257	-3.0	13.7	-27.1	312	-2.2	263	-	19.2	-
Vacuum (control)	337	5.6	263	-	14.2	-24.4	319	-	265	-	18.8	-

Percent changes relative to control (vacuum exposure) at 25°C; (2 sigma = ± 1.8 kcal/mole) ± ± 9.5% Δ E_a; (2 sigma = ± 0.3 sec) ± ± 2°K(T)⁻¹ ± 0.8% ΔT_E.

TABLE 3

Percent changes in explosion temperature and activation energy of PbN_6
exposed to vapors and pretreated at 25° and 74° for 72 hours

Vapor Exposure	PbN ₆ Pretreated at 74°C for 72 Hours					PbN ₆ Pretreated at 25°C for 72 Hours				
	t = 1 sec		t = 5 sec		E _a (kcal/mole)	t = 1 sec		t = 5 sec		E _a (kcal/mole)
	T _E (°C)	%ΔT _E	T _E (°C)	%ΔT _E		T _E (°C)	%ΔT _E	T _E (°C)	%ΔT _E	
aq-Freon	393	4.5	329	-	19.7	380	1.1	332	-	26.6
H ₂ O	407	8.2	333	-	18.8	373	-	330	-	27.5
Freon	403	7.2	341	3.3	21.6	375	-	341	3.3	42.1
Vacuum (control)	384	2.1	327	-	22.4	376	-	330	-	28.4

Percent changes relative to control (vacuum exposure) at 25°; (2 sigma = ± 1.8 kcal/mole) ≡ ± 6.3% ΔE_a; (2 sigma = ± 0.3 sec) ≡ ± 0.6% ΔT_E.

Percent changes in explosion temperature and activation energy of PbN_6/RDX exposed to vapors pretreated at 25° and 74° for 72 hours

Vapor Exposure	PbN ₆ /RDX Pretreated at 74°C for 72 Hours					PbN ₆ /RDX Pretreated at 25°C for 72 Hours						
	t = 1 sec T _E (°C)	%ΔT _E	t = 5 sec T _E (°C)	%ΔT _E	E _a (kcal/mole)	%ΔE _a	t = 1 sec T _E (°C)	%ΔT _E	t = 5 sec T _E (°C)	%ΔT _E	E _a (kcal/mole)	%ΔE _a
aq-Freon	306	—	242	-2.8	15.1	—	286	-6.5	246	—	22.9	35.5
H ₂ O	348	13.7	264	6.0	11.9	-29.6	304	—	244	-2.0	16.0	—
Freon	310	1.3	252	—	16.9	—	295	-3.6	248	—	20.2	19.5
Vacuum (control)	315	2.9	255	2.4	16.0	—	306	—	249	—	16.9	—

Percent changes relative to control (vacuum exposure) at 25°C; (2 sigma = ± 1.8 kcal/mole) $\bar{z} \pm 10.6\% \Delta E_a$; (2 sigma = ± 0.3 sec) $\bar{z} \pm 0.6\% \Delta T_F$.

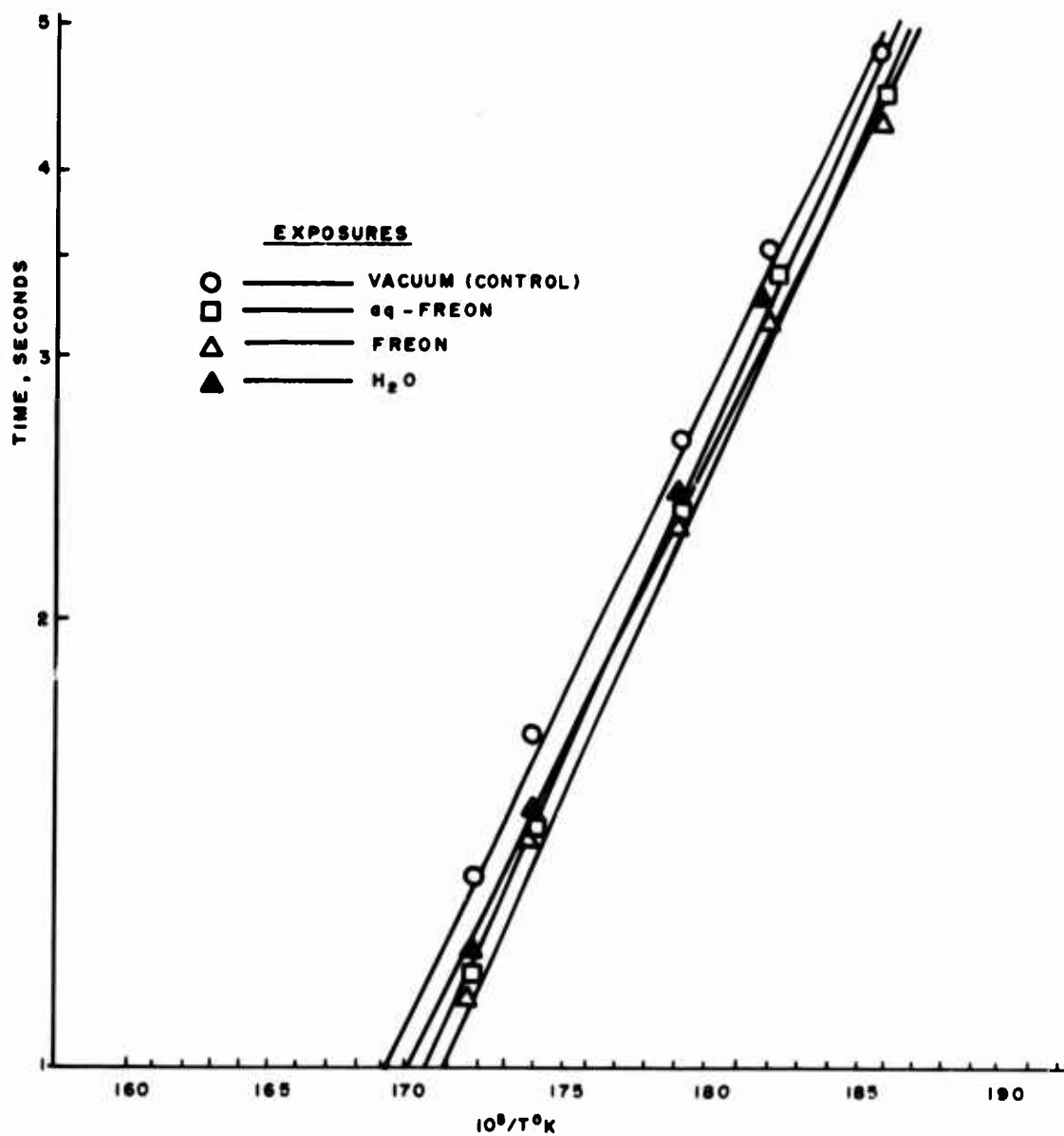


Fig 1 Sensitivity of RDX to thermal explosive decomposition as a function of various exposures at 25°C for 72 hours

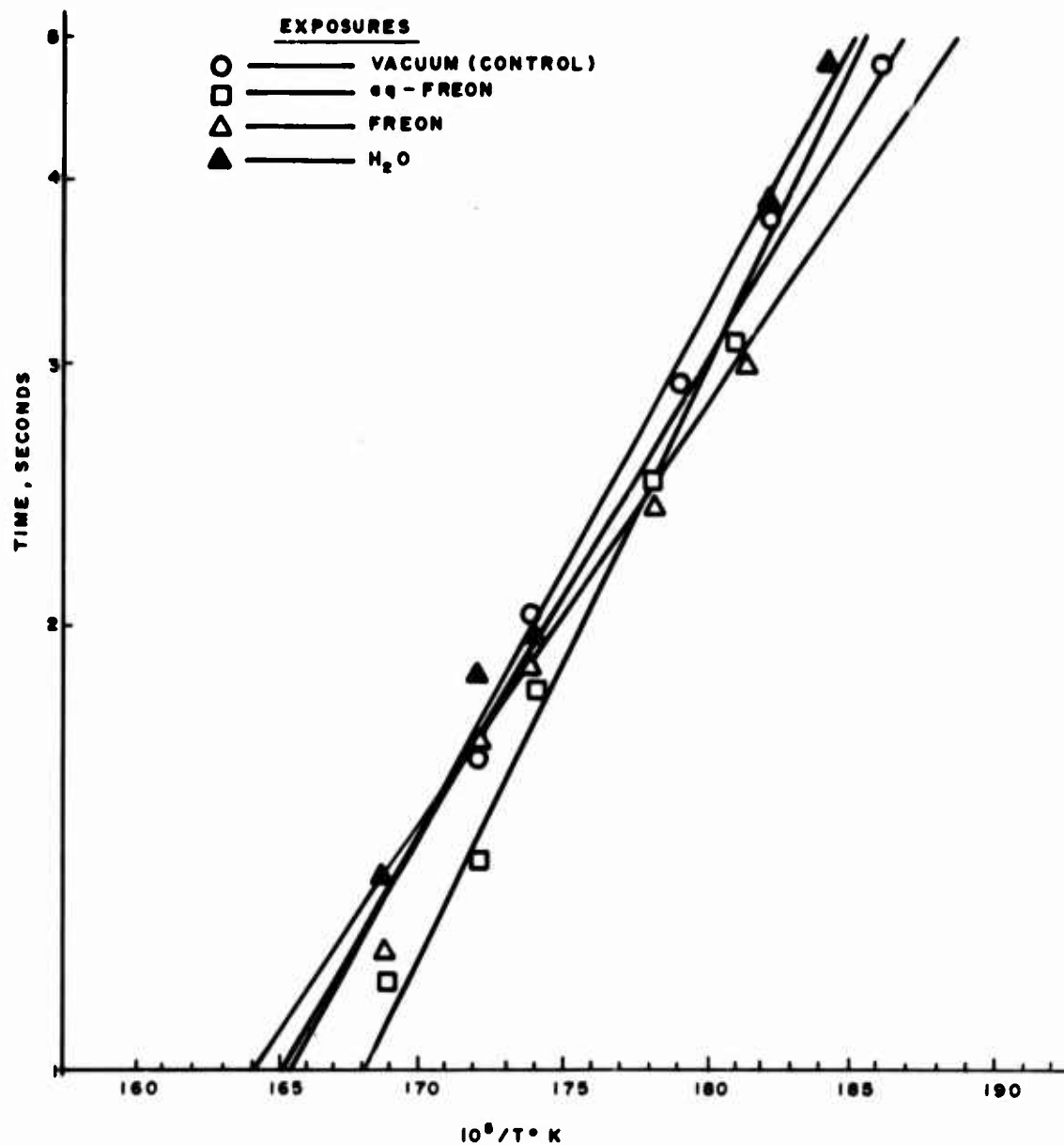


Fig 1A Sensitivity of RDX to thermal explosive decomposition as a function of various exposures at 74°C for 72 hours

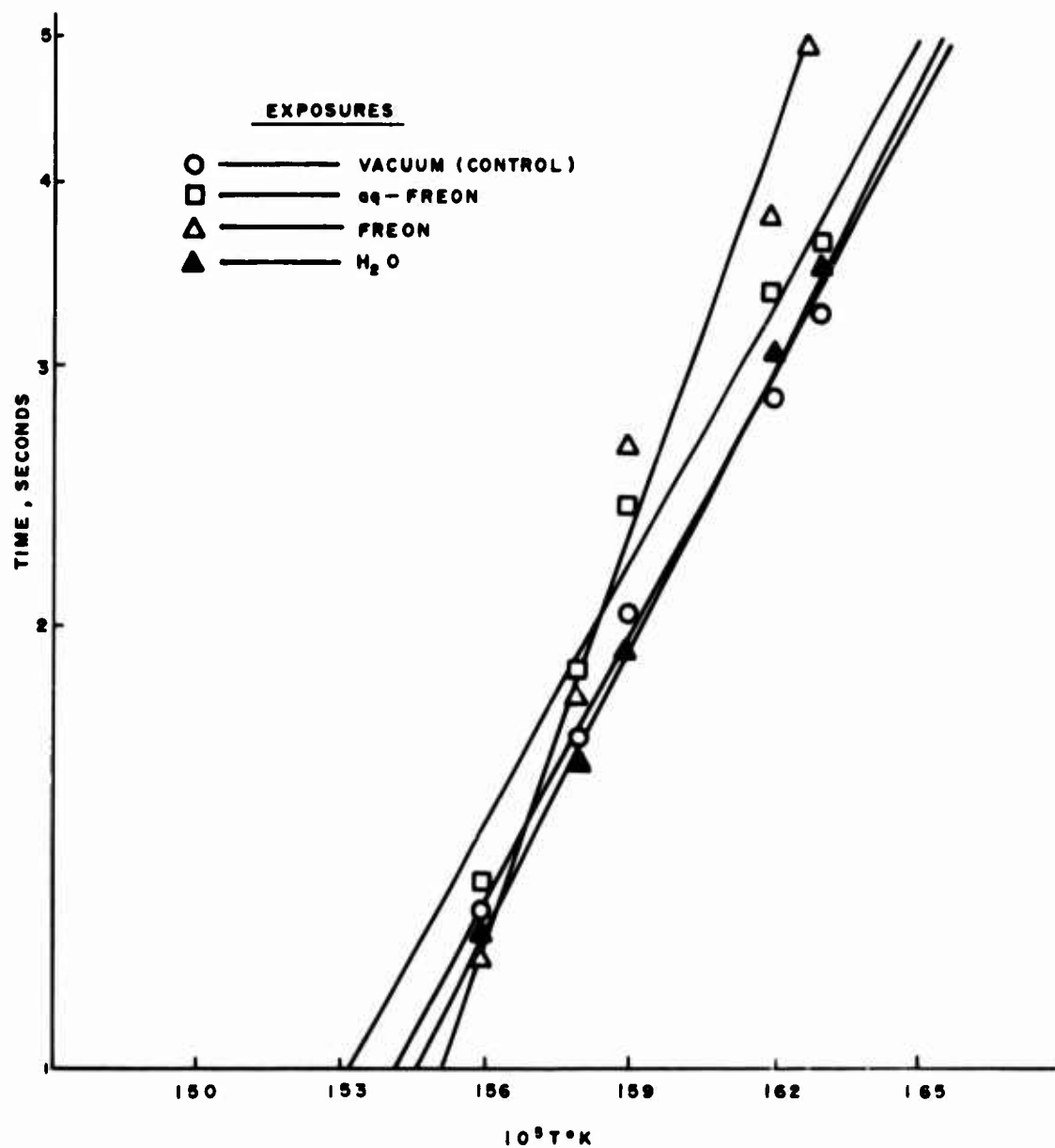


Fig 2 Sensitivity of PbN₆ to thermal explosive decomposition as a function of various exposures at 25°C for 72 hours

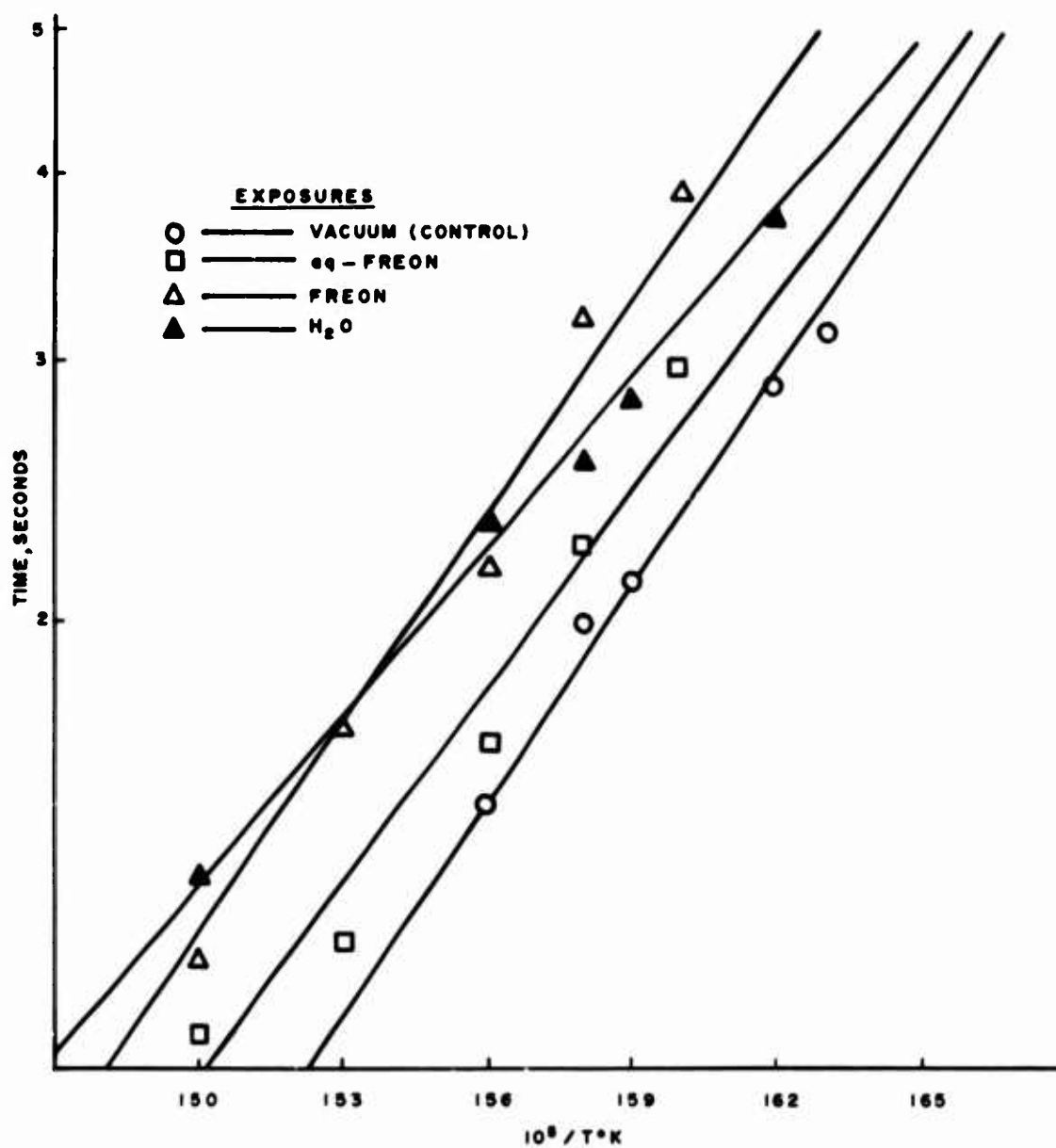


Fig 2A Sensitivity of PbN_6 to thermal explosive decomposition as a function of various exposures at 74°C for 72 hours

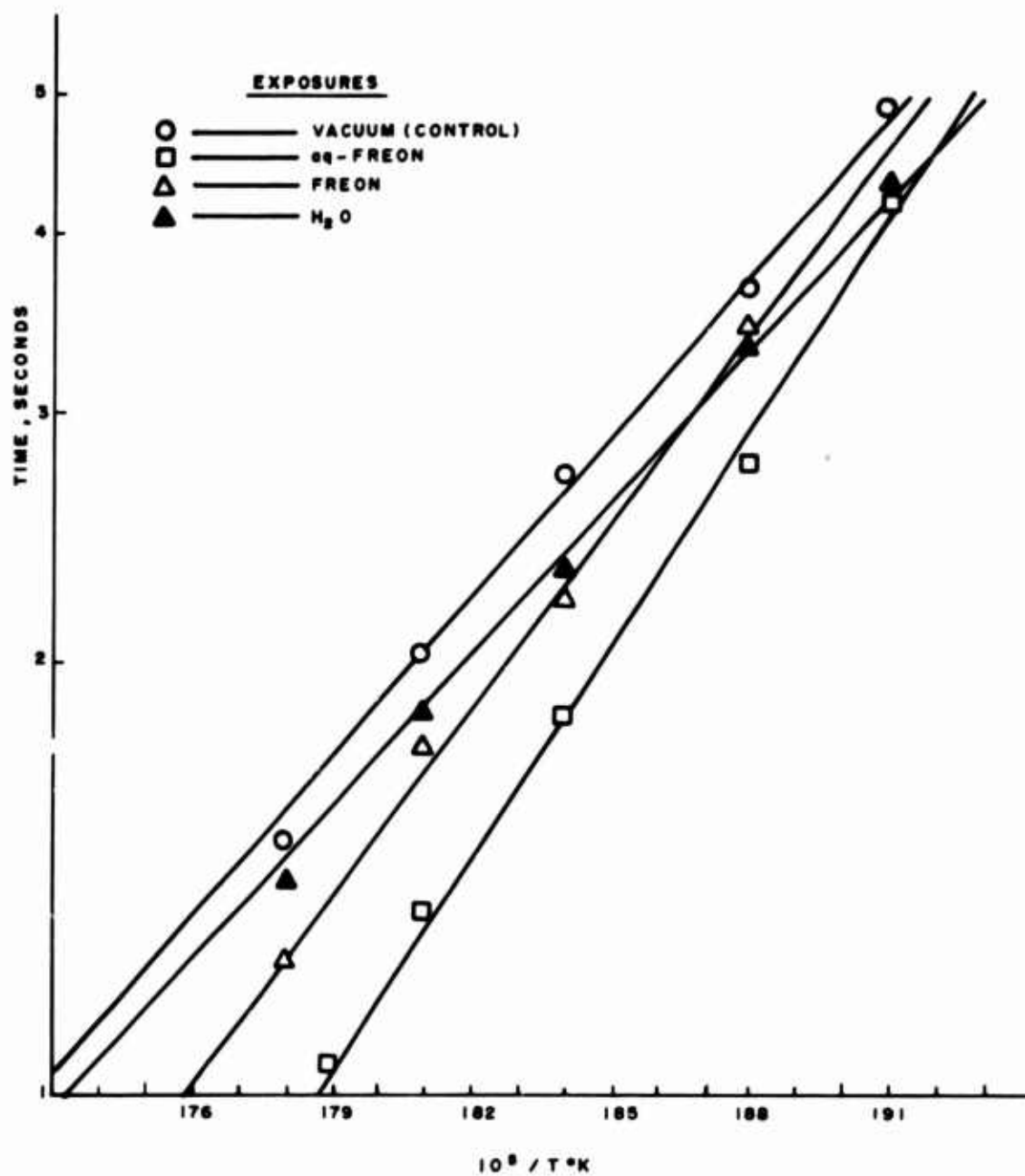


Fig 3 Sensitivity of PbN₆/RDX to thermal explosive decomposition as a function of various exposures at 25°C for 72 hours

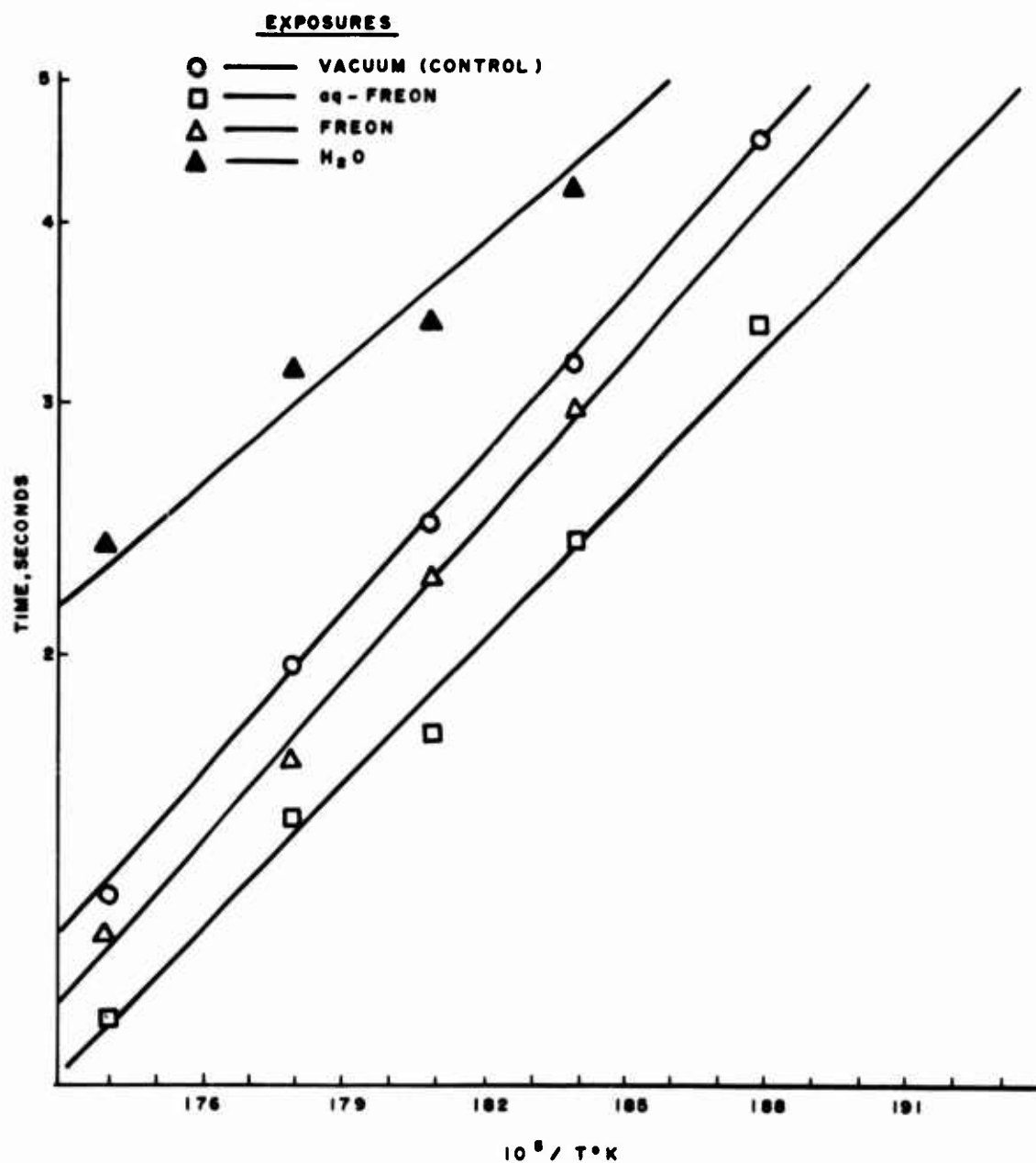


Fig 3A Sensitivity of PbN₆/RDX to thermal explosive decomposition as a function of various exposures at 74°C for 72 hours

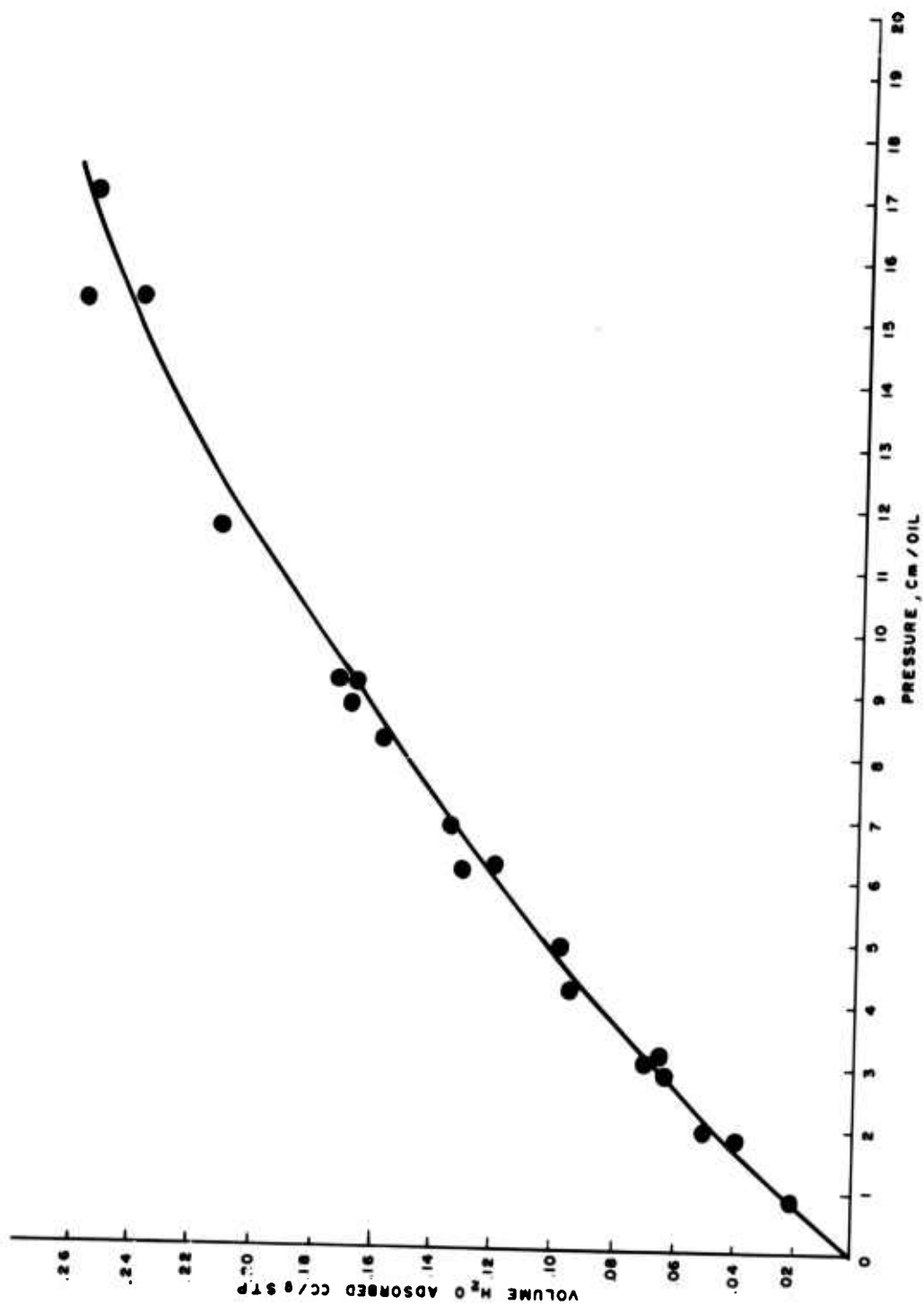


Fig 4 Adsorption isotherm, H₂O on RDX at 25°C

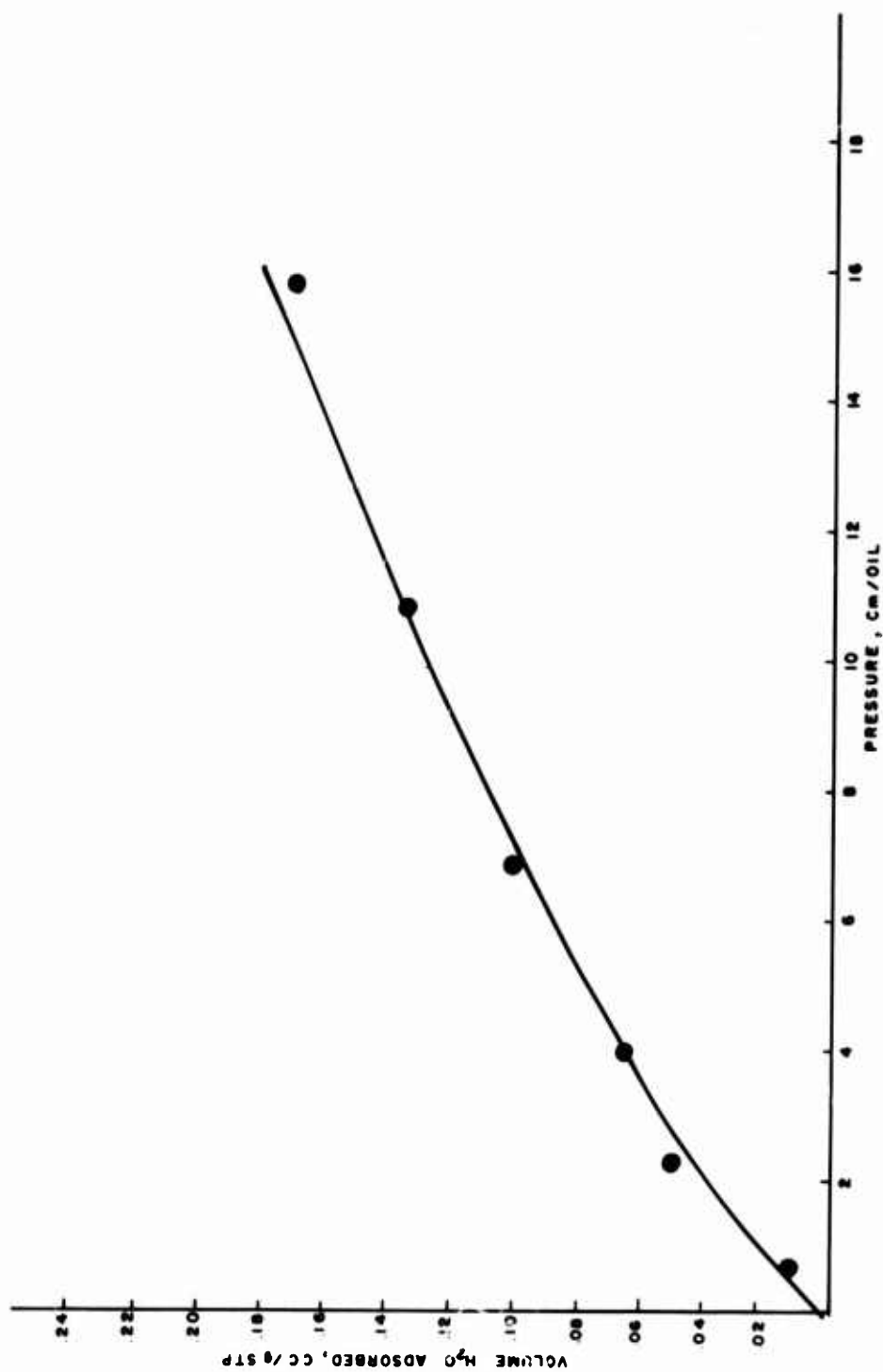


Fig 5 Adsorption isotherm, H₂O on PbN₆ at 25°C

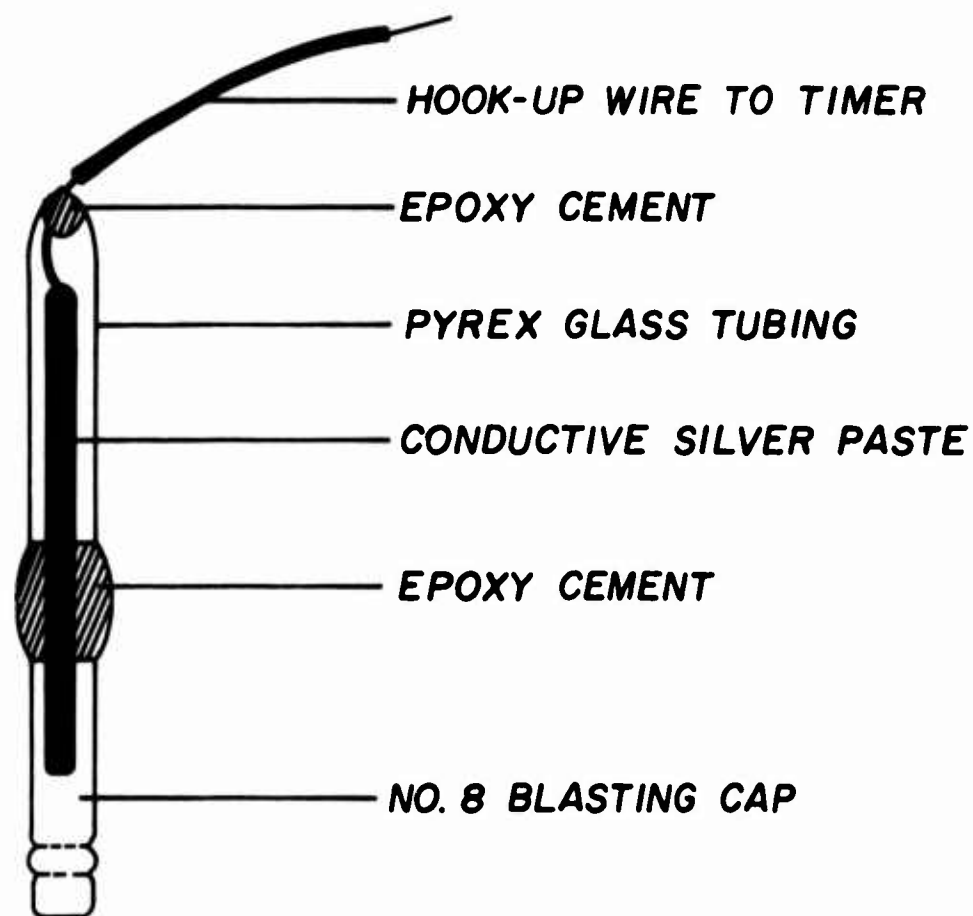


Fig 6 Assembly unit for the containment of vapors in contact with the explosive during the induction period of thermal explosive decomposition

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13. ABSTRACT The Picatinny Arsenal explosion temperature test has been modified to study the effect produced on the induction period by vapors in contact with the surface of explosives. The test has been found to be reproducible and significantly responsive to changes in times to explosion and activation energy of RDX, PbN ₆ , and RDX/PbN ₆ mixture exposed to H ₂ O and Freon vapors. These changes are postulated to be a function of vapor-surface interactions which correspondingly alter the rate of surface heat dissipation and mechanism of chemical reactions which initiate explosive decomposition.			

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Thermal sensitivity						
RDX						
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Explosive decomposition						
Activation energy						
Vapor-surface interaction						
Initiation of explosives						
Time to explosion						
Activation energy						